Plastic Deformation at Micron-Scale in Cu Thin Films and Lines Studied by X-ray Microdiffraction

R. Spolenak[2], N. Tamura[1], B.C. Valek[3], R.S. Celestre[1], A.A. MacDowell[1], H.A. Padmore[1] and J.R. Patel[1,4]
[1]Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA
[2]Bell Laboratories, Lucent Technologies, Murray Hill NJ 07974 USA
[3] Dept. Materials Science & Engineering, Stanford University, Stanford CA 94305 USA
[4]SSRL/SLAC, Stanford University, P.O.BOX 43459, Stanford CA 94309 USA

INTRODUCTION

Plasticity in thin films has been extensively studied by averaging techniques such as wafer curvature, tensile testing, bulge testing and conventional x-ray diffraction, but is still only partially understood. In the present work, we have investigated plastic phenomena using the X-Ray Microdiffraction Laue technique on beamline 7.3.3. at the ALS. This technique is able to determine the full strain/stress tensor (6 components) and the orientation matrix at submicron scale in thin films with an accuracy of about $2\ 10^{-4}$ in strain and less than 0.1º in orientation. In contrast to the complementary technique of electron microscopy, no sample preparation is necessary (i. e. the strain/stress state remains unaltered). Local plastic deformation of single grains in a sputtered Cu blanket film was studied between room temperature and 225 ºC. Furthermore we measured effects of two-dimensional confinement of passivated electroplated Cu damascene lines at temperature varying from room temperature to 275 ºC.

EXPERIMENT

The experimental setting for the X-ray Microdiffraction Laue technique is described elsewhere [1,2]. The sputtered Cu thin film sample has a thickness of 1.5 µm. An area of 15x15 µm has been scanned with a submicron sized white beam and a step size of 1 µm to obtain orientation and deviatoric stress maps at different temperature during a thermal cycle between 25 and 225 °C. Similar measurements have been carried out on encapsulated damascene Cu lines of length 20 µm, thickness 0.8 µm and different widths (5, 2 and 0.8 µm). The entire lines were scanned during a temperature cycle between 25 and 275 °C.
At each step, a diffraction pattern was collected using a large area CCD camera. The position of the CCD with respect to the incoming beam and the sample was calibrated with a precision of about 10 µm using the single crystal Laue pattern coming from the Si wafer under the thin films.

RESULTS AND DISCUSSION

Fig. 1 compares an image obtained by FIB (Focused Ion Beam) with orientation maps obtained by X-ray microdiffraction at room temperature. The FIB offers two orders of magnitude better spatial resolution than X-ray microdiffraction but the latter technique provides unique quantitative information such as accurate crystal orientation and strain/stress tensor values [3, 4]. The stress data obtained by X-ray microdiffraction was also compared with values obtained on the same sample by wafer curvature measurements. Both techniques show that the film is in average under biaxial tensile stress at room temperature. However, X-ray microdiffraction gives additional information. It indicates that the stress distribution is actually very broad and that at the micron scale the stress is in fact triaxial, rather than biaxial.

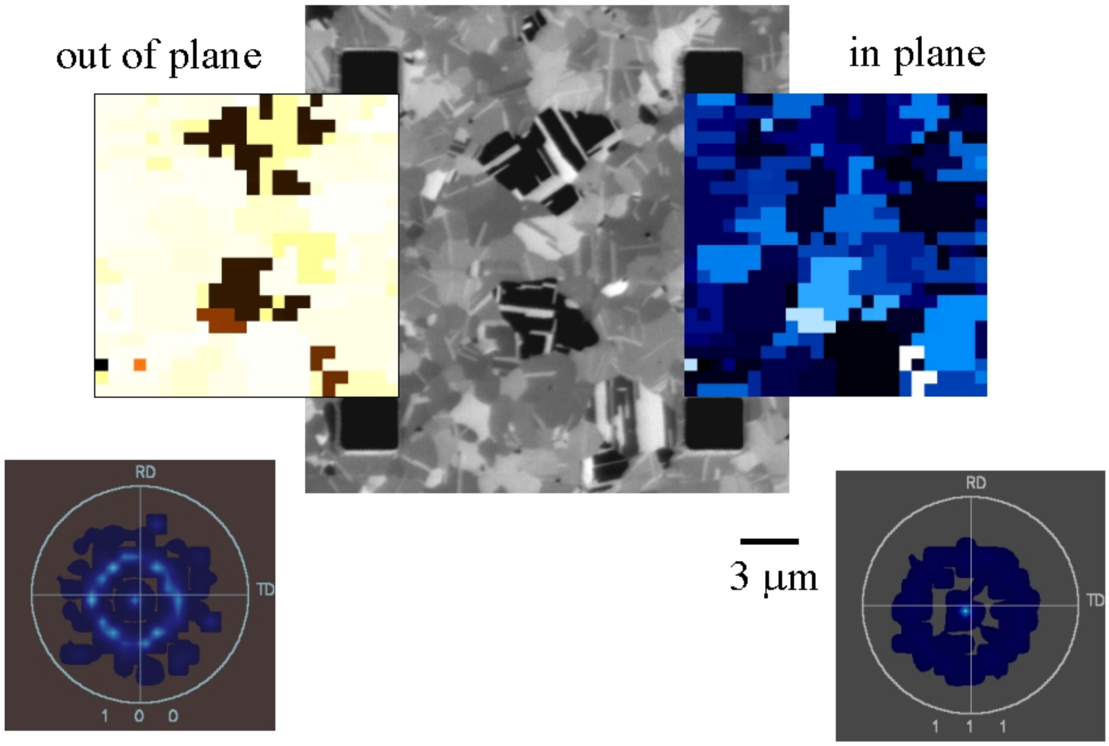

Fig 1.- Out of plane (left) and in-plane (right) orientation maps obtained by the X-ray Microdiffraction Laue technique. The maps are compared with an FIB image (center). (001) grains with their twins platelets are clearly visible in the out-of-plane map (in black) as well as in the FIB image. Bottom left and right are respectively the (100) and (111) pole figures within the 15x15 μm scanned area.

Some local areas in the film are even in the compressive regime. During heating, the stress decreases and becomes compressive. The stress-temperature curve during heating departs from the so-called "thermoelastic" behavior even before the average of the stress goes compressive. X-ray microdiffraction provides a simple explanation to this effect: grains, which were already compressive, will have already reached their yield stress.

Mechanical properties of thin films differ significantly from bulk materials due to the particular confinement geometry. For instance, the yield stress in thin metallic films is usually higher than the bulk value. Lines offer an additional degree of confinement and their mechanical properties are expected to be different as well [5]. X-ray microdiffraction measurements on individual lines (Fig. 2) shows that with decreasing line width, the stress goes from a biaxial to a triaxial state. The area of hysteresis during a temperature cycle is an indication of the amount of plastic deformation. This area decreases with the line width and the narrowest line does not show plasticity within the resolution of the experiment.

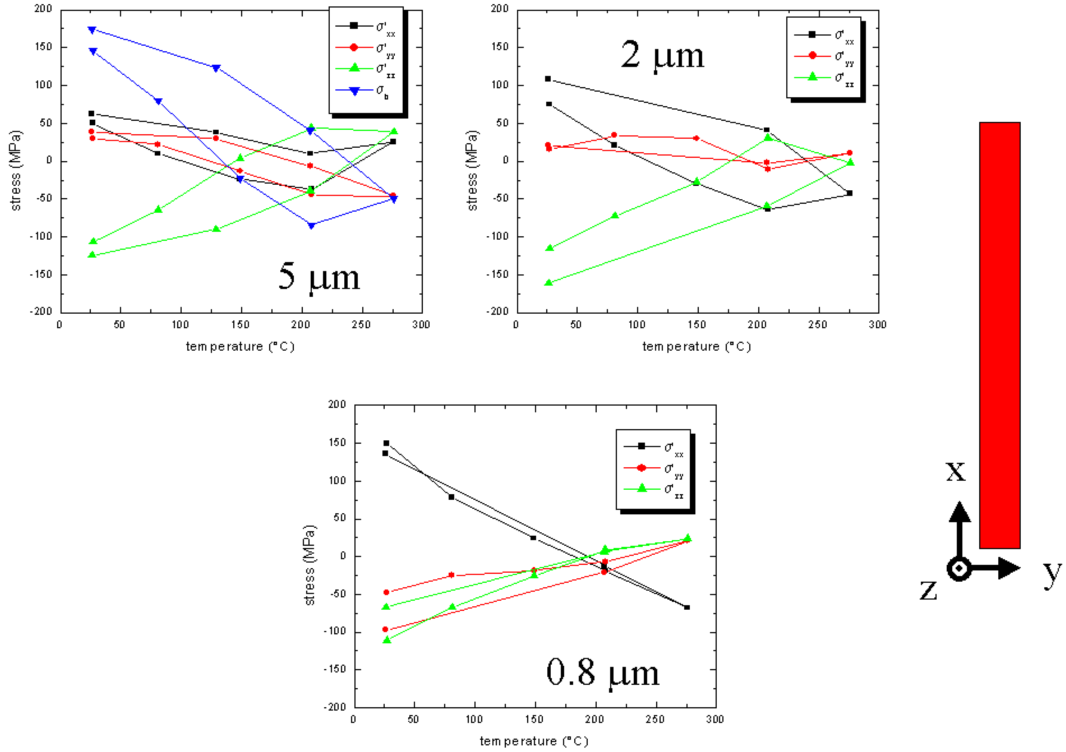

Fig 2.- Stress-temperature curves for 20 µm damascene Cu lines of width 5 µm (top left), 2 µm (top right) and 0.8 µm (bottom). Black , red and green curves represent respectively the deviatoric stress component along (x), across (y) and normal to the surface (z) of the line. The blue curve for the 5 µm line is the average "biaxial" stress.

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Principal investigator: Nobumichi Tamura, Advanced Light Source, Lawrence Berkeley National Laboratory. Email: ntamura@lbl.gov. Telephone: 510-486-6189.